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PATENT & TRADEMARK OFFICE

Serial No.: 09/235,686

PATENT

IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

In re Application of:	
Ann Xiaoan Liu, et al.	Docket: ACO6105PDUS
Serial No.: 09/235,686 Filing Date: January 22, 1999	Examiner: M. Jackson
	Group Art Unit: 1773
For: SYNTHETIC RESIN FILM FOR LAMINATES AND METHOD OF PRODUCING SAME	

Assistant Commissioner for Patents
Washington, D.C. 20231

RECEIVED
FEB 0 8 2002
TC 1700

DECLARATION UNDER 37 CFR 1.132

I, Ann Xiaoan Liu, do hereby declare as follows:

1. I am the technical Manager at Casco Impregnated Papers Inc., Akzo Nobel, Cobourg, Ontario, where I have worked for 6 years.

2. I conducted and/or had control over the following comparative examples, Sample ID 1-5, in Table A, below. These comparative examples were made to show the importance of the substantially spherical nature of the low profile additive according to the present invention.

To exemplify this substantially spherical shape and contrast it with other types of additives, attached are copies of pictures (1) Zeeospheres ceramic microspheres, which have a substantially spherical shape, and calcium carbonate, mica and wollastonite, which have jagged edges and do not have a substantially spherical shape, (2) c llulose fiber, which have a fibrous shape and

do not hav a substantially spherical shape, and (3) Alumina (Al_2O_3) (7 slides), which have jagged edges and do not have a substantially spherical shape.

In the examples of the invention, e.g. Sample Nos. 1-14 in Table 1 on page 9 of the specification, the presence of the low profile additive increases the scratch resistance compared to the scratch resistance in the absence of the low profile additive. Comparative examples, Sample ID 1-5, in Table A include an additive which is not substantially spherical, in contrast to the low profile additive of the present invention. The additive in Table A is alumina. As a result of using this additive, the scratch resistance is the same or worse compared to the scratch resistance in the absence of the additive.

Table A
Scratch resistance test results
using an additive which is not substantially spherical (alumina)

Sample ID	Paper basis weight g/m2	Film weight g/m2	Additiveg/m2	Additive %	Scratch with additive	Scratch without additive
1	70	205	5.7	4.2	2.5	2.5
2	80	218	7.1	5.1	2.5	3.0
3	85	225	4.5	3.2	3.0	3.0
4	83	215	4.7	3.6	2.5	2.5
5	105	285	6.5	3.6	2.0	2.0

3. I have determined the percentage of low profile additive in Sample Nos. 1-14, shown in Table 1 on page 9 of the specification, by using the disclosed amount of additive, the disclosed paper basis weight and the known typical film weight of treated decorative papers, as disclosed in the attached 1998 TAPPI Proceedings Plastic Laminates Symposium, August 17-20, 1998 ("TAPPI Proceedings"). According to TAPPI Proceedings, the "papers typically range from 80 to 115 grams per square met r, with an overall treated weight of 200 to 350 grams per square meter." TAPPI Proceedings at 76. These typical weights of the

paper and treated paper are used to determine the resulting typical range of resin treatment percentage using the formula in TAPPI Proceedings at 77, which, in turn, is used to determine the film weight and, ultimately, the percentage of low profile additive in Sample Nos. 1-14.

The formula (TAPPI Proceedings at 77) calculates the weight of treated paper by dividing the basis weight of the paper prior to treating by the inverse of the resin treatment percentage (basis weight of untreated paper/inverse of resin treatment percentage = basis weight of treated paper) . Thus, to determine the typical range of resin treatment percentages, I divided the basis weight of untreated paper by the basis weight of treated paper, which yielded the inverse of the resin treatment percentage, and then took its inverse.

Using the range of weights for treated and untreated papers and the formula, I determined that the typical range of resin treatment percentages is 60-66%. My calculations are shown below:

80/200 = 0.40 (the inverse of the resin treatment percentage is 40%); Thus, the resin treatment percentage is 60%.

115/350 = 0.33 (the inverse of the resin treatment percentage is 33%); Thus, the resin treatment percentage is 66%.

Based on the typical range of resin treatment percentages being 60-66%, I calculated the film weight of each of Sample Nos. 1-14 (in accordance with the paper basis weight of each of these samples) at 60% and at 66% and I used this data to calculate the % of Additive = additive(g/m^2) X 100 /(Film weight – Paper basis weight). The results of these calculations are shown in Tables B and C below.

Table B; 60 % resin content

Sample #	Paper basis wt.	Film wt.	Additive g/m2	Additiv %	Scratch with additive	Scratch without additive
1	80	200.0	6.8	5.67	4.0	2.5
2	85	212.5	0.7	0.56	2.5	2.0
3	85	212.5	5.0	3.92	3.0	2.0
4	72	180.0	5.9	5.46	3.5	3.0
5	83	207.5	2.7	2.19	4.0	3.5
6	85	212.5	2.1	1.68	3.0	2.5
7	85	212.5	3.0	2.36	3.5	3.0
8	75	187.5	5.2	4.62	3.5	3.0
9	80	200.0	5.7	4.75	4.5	3.5
10	105	262.5	5.6	3.56	3.5	2.0
11	80	200.0	5.9	4.92	3.5	2.0
12	80	200.0	6.5	5.42	4.0	2.5
13	130	325.0	3.7	1.90	4.5	3.0
14	80	200.0	3.3	2.75	3.8	3.0

Table C; 66% resin content

Sample #	Paper basis wt.	Film wt.	Additive g/m2	Additive %	Scratch with additive	Scratch without additive
1	80	235.3	6.8	4.38	4.0	2.5
2	85	250.0	0.7	0.43	2.5	2.0
3	85	250.0	5.0	3.03	3.0	2.0
4	72	211.8	5.9	4.22	3.5	3.0
5	83	244.1	2.7	1.69	4.0	3.5
6	85	250.0	2.1	1.30	3.0	2.5
7	85	250.0	3.0	1.82	3.5	3.0
8	75	220.6	5.2	3.57	3.5	3.0
9	80	235.3	5.7	3.67	4.5	3.5
10	105	308.8	5.6	2.75	3.5	2.0
11	80	235.3	5.9	3.80	3.5	2.0
12	80	235.3	6.5	4.19	4.0	2.5
13	130	382.4	3.7	1.47	4.5	3.0
14	80	235.3	3.3	2.13	3.8	3.0

I declare that all statements made herein of my own knowledge are true and that all statements made on information and belief are believed to be true; and further that these statements were made with the knowledge that willful false statements and the like so made are punishable by fine or imprisonment, or both,

under Section 1001 of Title 18 of the United Stated Code and that such willful false statements may jeopardize the validity of this application or any patent resulting therefrom.

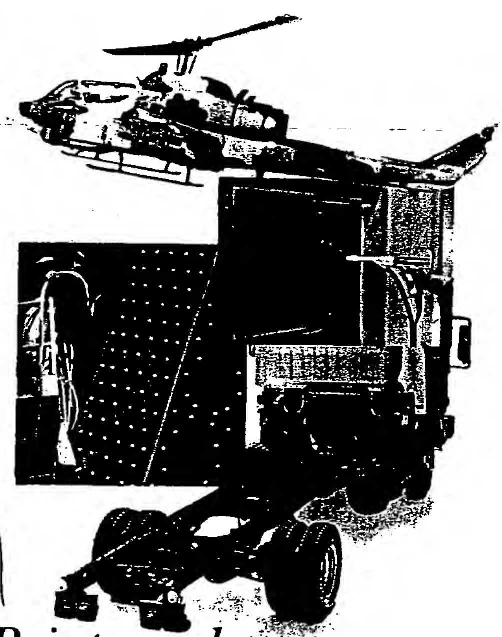
DATE: Nov 6 /00

BY:
Name: Ann Xiaoan Liu
Title: Technical Manager

3M

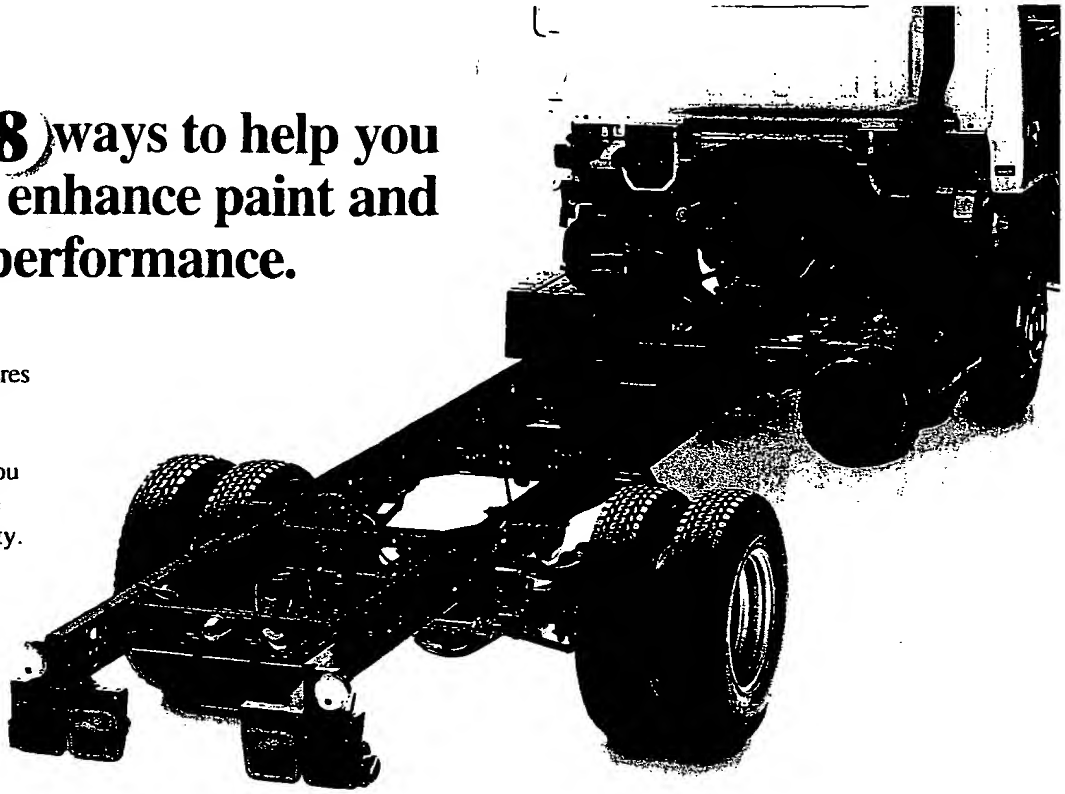
Zeeospheres™

Ceramic Microspheres

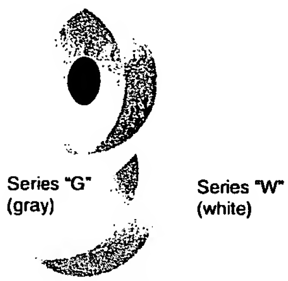


Paints and Powder Coatings Applications Profile

Engineered for 8 ways to help you reduce costs and enhance paint and powder coating performance.



3M™ Zeeospheres Ceramic Microspheres are high strength, inert fine particles with intrinsic hardness. Zeeospheres microspheres are engineered to help you reduce costs, increase solids, enhance properties, and improve processability.



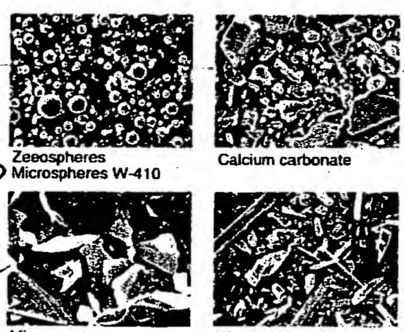
Spherical simplicity to help meet complex challenges.

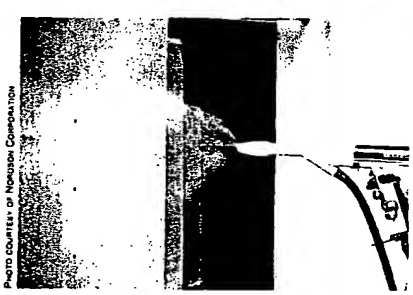
Typical application – truck under-carriage coating that combines lower VOCs, high solids, and corrosion resistance (see 5).

1 Lower viscosity and improved flow

Unlike many irregularly shaped fillers, Zeeospheres microspheres roll easily over one another, similar to ball bearings. This contributes to lower viscosity, better flow, and improved sprayability.

Shape of different material

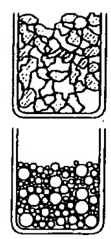




Typical application – powder coating with spherical dispersion that improves material handling for consistently smooth surfaces.

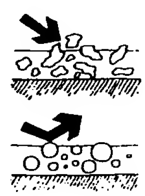
2 Higher filler loading to reduce cost

With the lowest surface area to volume ratio of any shape, microspheres reduce resin demand and increase volume loading capacity. Smaller spheres fill voids between larger ones to enhance packing for higher solids/lower VOCs, and reduced cost.



3 Hardness and abrasion resistance

Mohs 7 hardness and spherical shape contribute to increased hardness and burnish/abrasion resistance of the finished surface. Surfaces stay new looking longer to save the time and cost of touch-ups or repainting. With ordinary fillers, soft or jagged particles on the surface break or wear away.



Typical application – metal surfaces of office furniture exposed to daily use and abuse.

Typical application – high scrub, crystalline silica-free interior paints that maintain optical qualities after repeated cleaning.

	ARBOCEL BSCH 200 H	ARBOCEL BSCH 200 D	ARBOCEL BSCH 750 H	ARBOCEL BSCH 750	ARBOCEL ZZ 8-2 CA 1	ARBOCEL II 8-2	ARBOCEL ZZ 8-1	ARBOCEL PZ 8
	rohweiß off-white	dunkel dark	rohweiß off-white	dunkel dark	grau grey	grau grey	grau grey	grau grey
	mittellange Faser medium fibre	mittellange Faser medium fibre	Langfaser long fibre	Langfaser long fibre	Langfaser long fibre	Langfaser long fibre	Langfaser long fibre	Langfaser long fibre
	200	200	650	650	1000	900	1100	1400
	25	25	25	25	45	40	45	45
	max. 98	max. 98	max. 98	max. 98	~50	~70	~80	~80
	~1	~1	~1	~1	~50	~25	~15	~15
	7±1	7±1	7±1	7±1	7,5±1	7,5±1	7,5±1	7,5±1
	100 - 140	100 - 140	40 - 70	40 - 70	60 - 100	30 - 50	20 - 40	10 - 25
	95	95	99	99	90	95	99	99
	40	40						
			50	50				
	0,5	0,5			20	40	60	70
			5	5				

Baumwollcellulose

cotton cellulose

Technische Rohcellulose

technical raw cellulose

Wie bei allen natürlichen Produkten sind geringfügige Schwankungen der angegebenen Kennwerte möglich.

As with all natural products slight differences to the scaled values may arise.

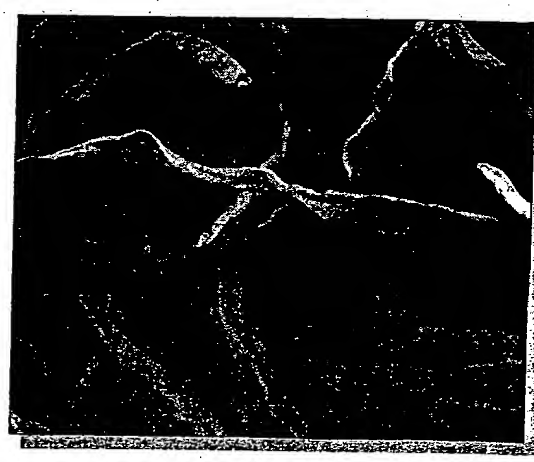
Shape of cellulose fiber

Struktur von ARBOCEL® Cellulosefasern

Structure of ARBOCEL® cellulose fibres

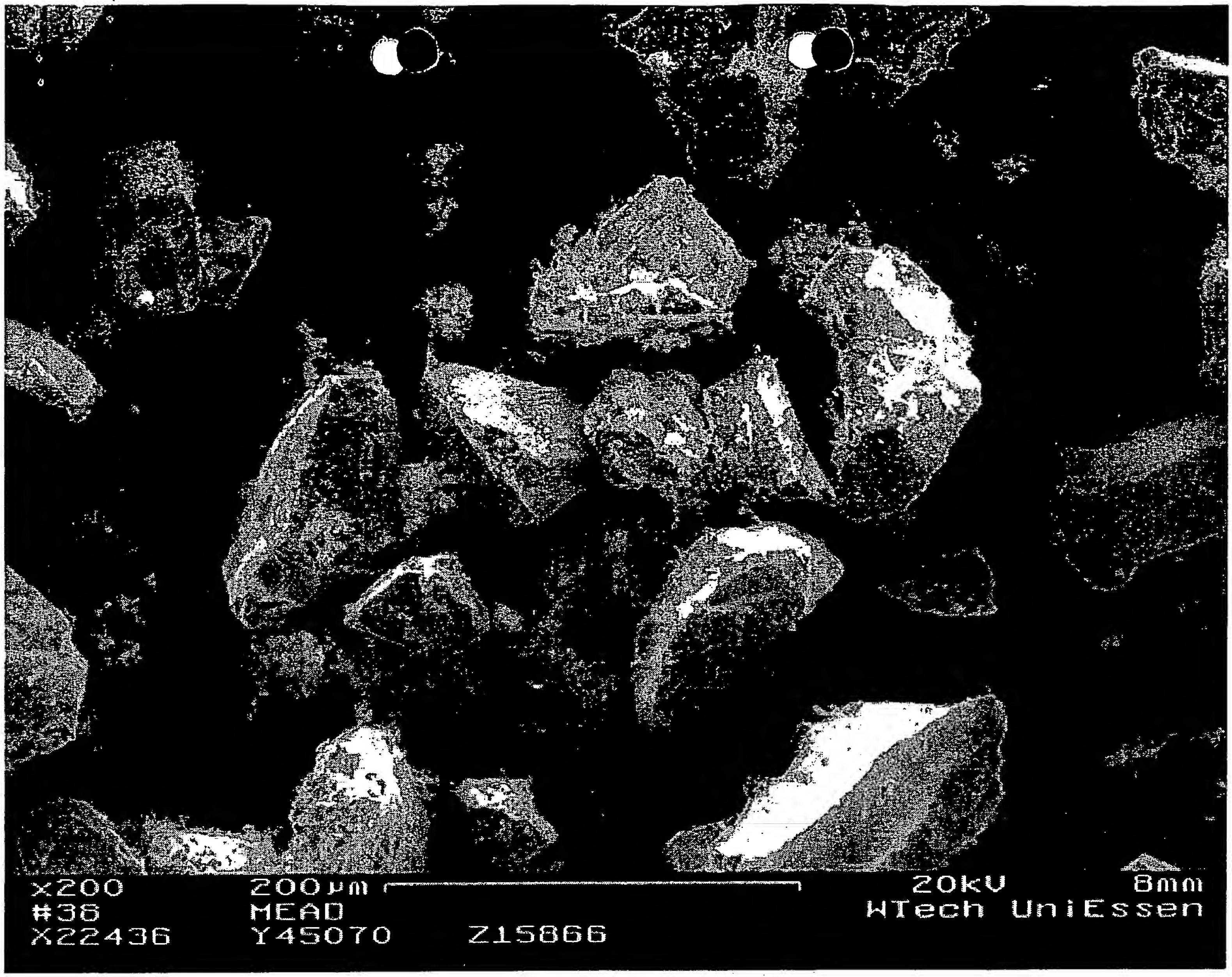
ARBOCEL BE 600 - 30 (x 300)

ARBOCEL BE 00 (x 300)



ARBOCEL B 400 (x 300)

Cellulose CAS - No. 9004-34-6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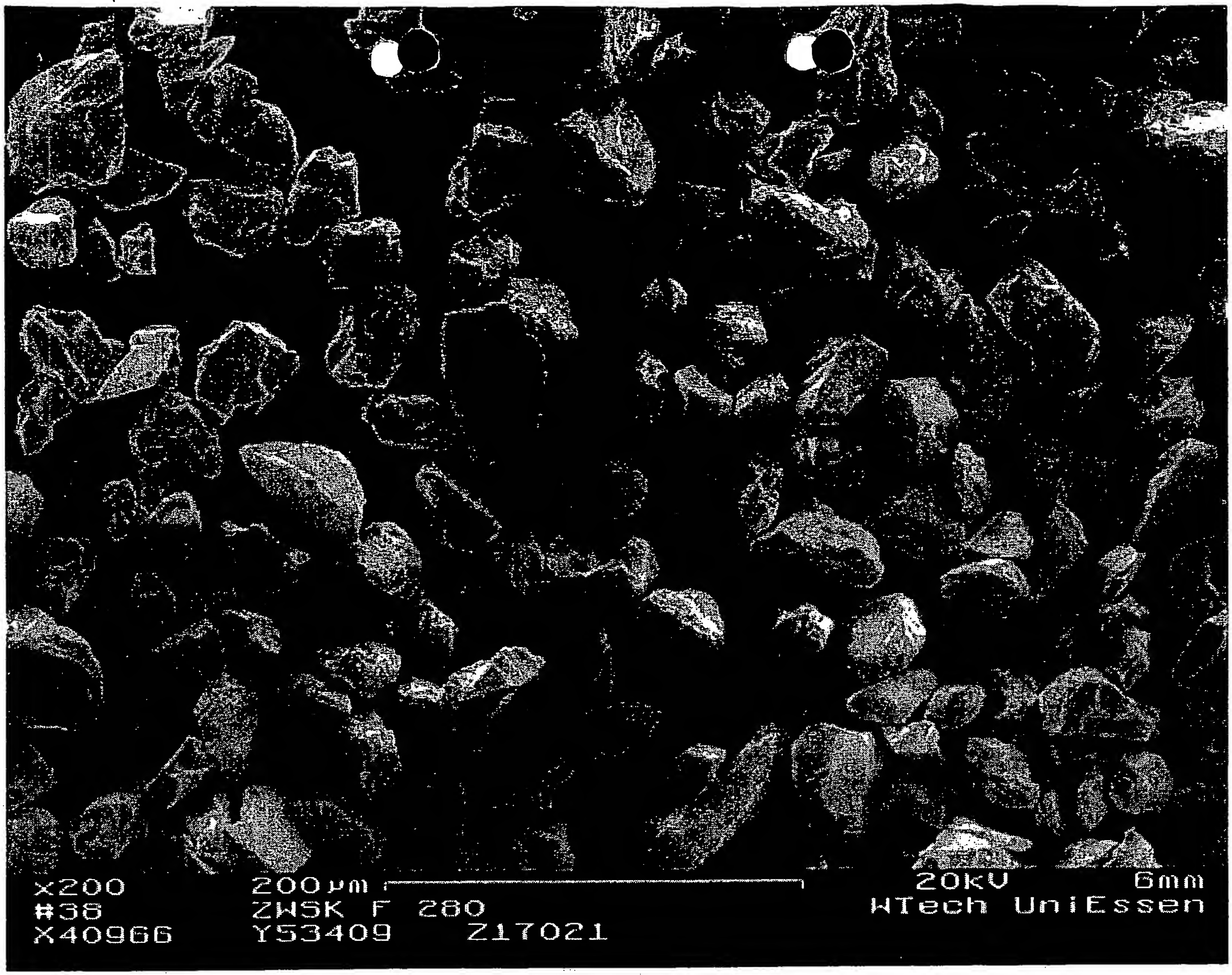
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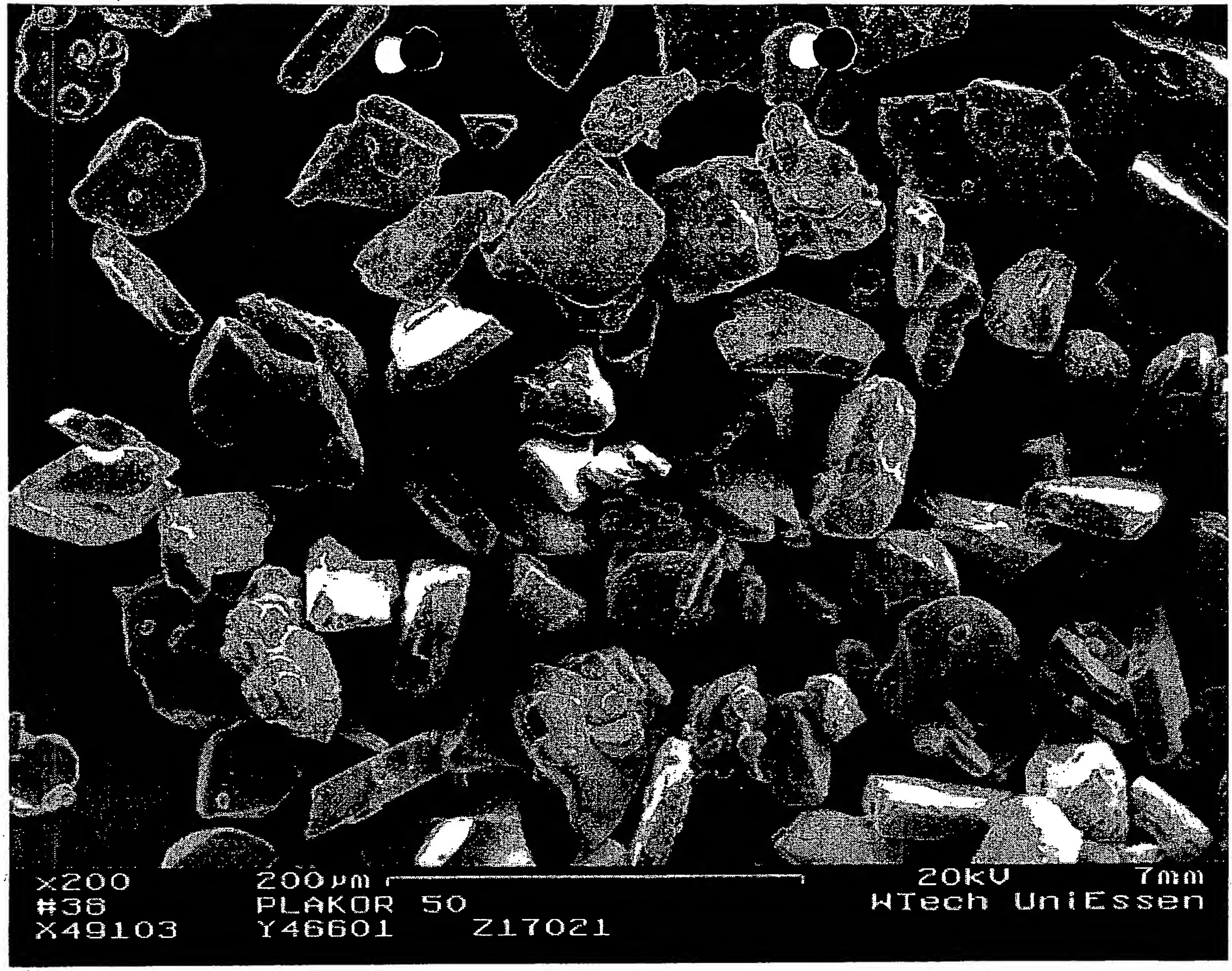
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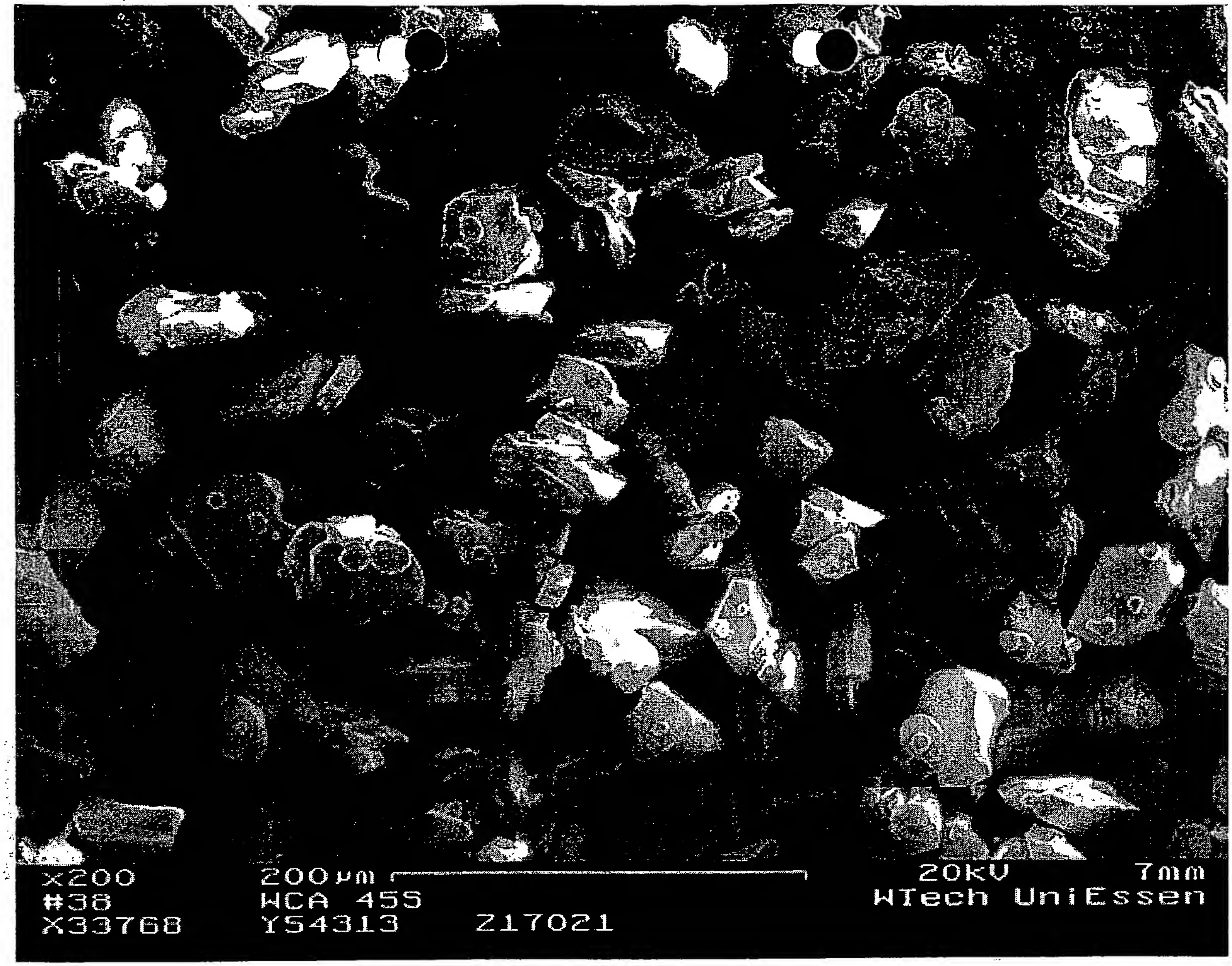
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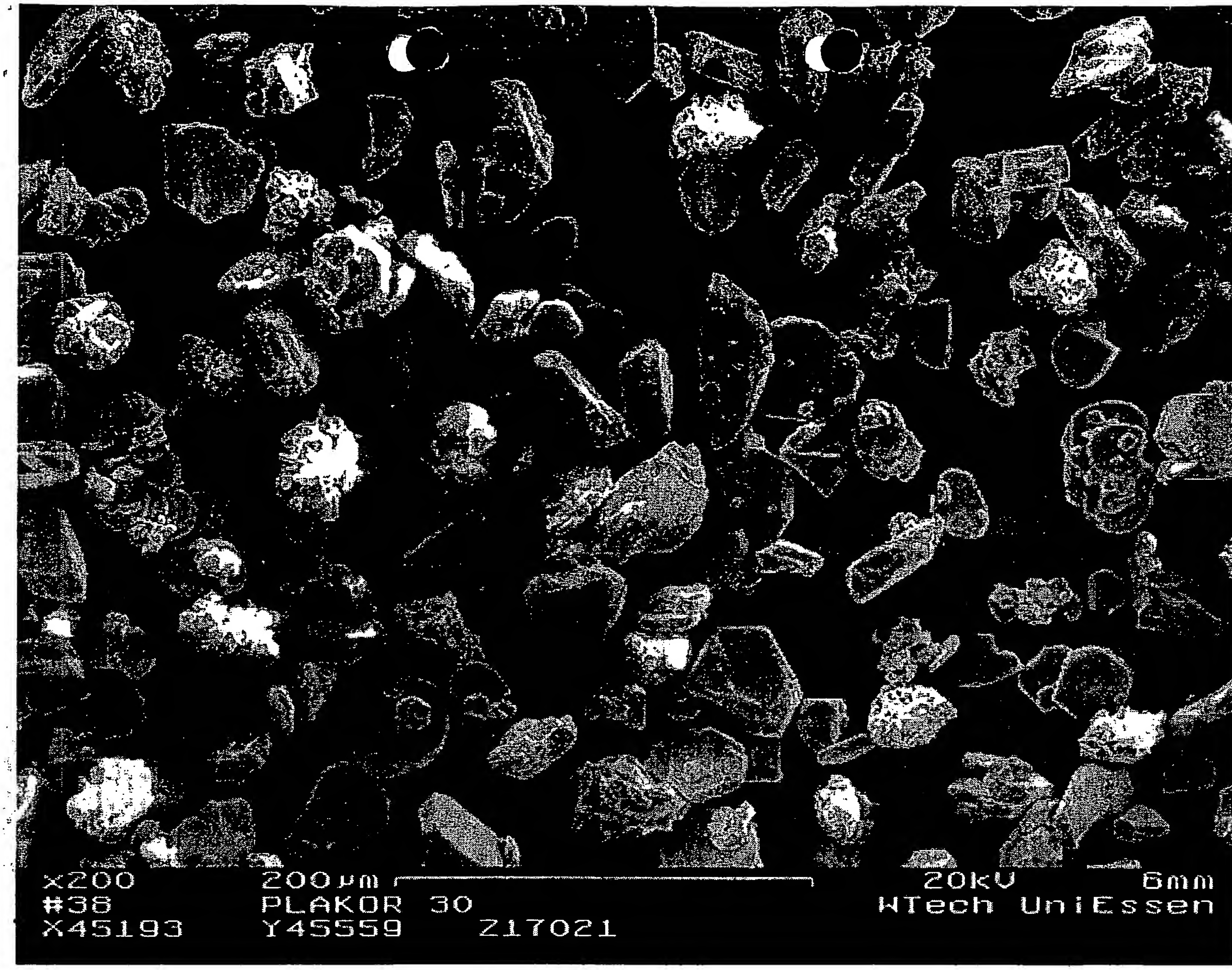
X38810 Y33446 Z17021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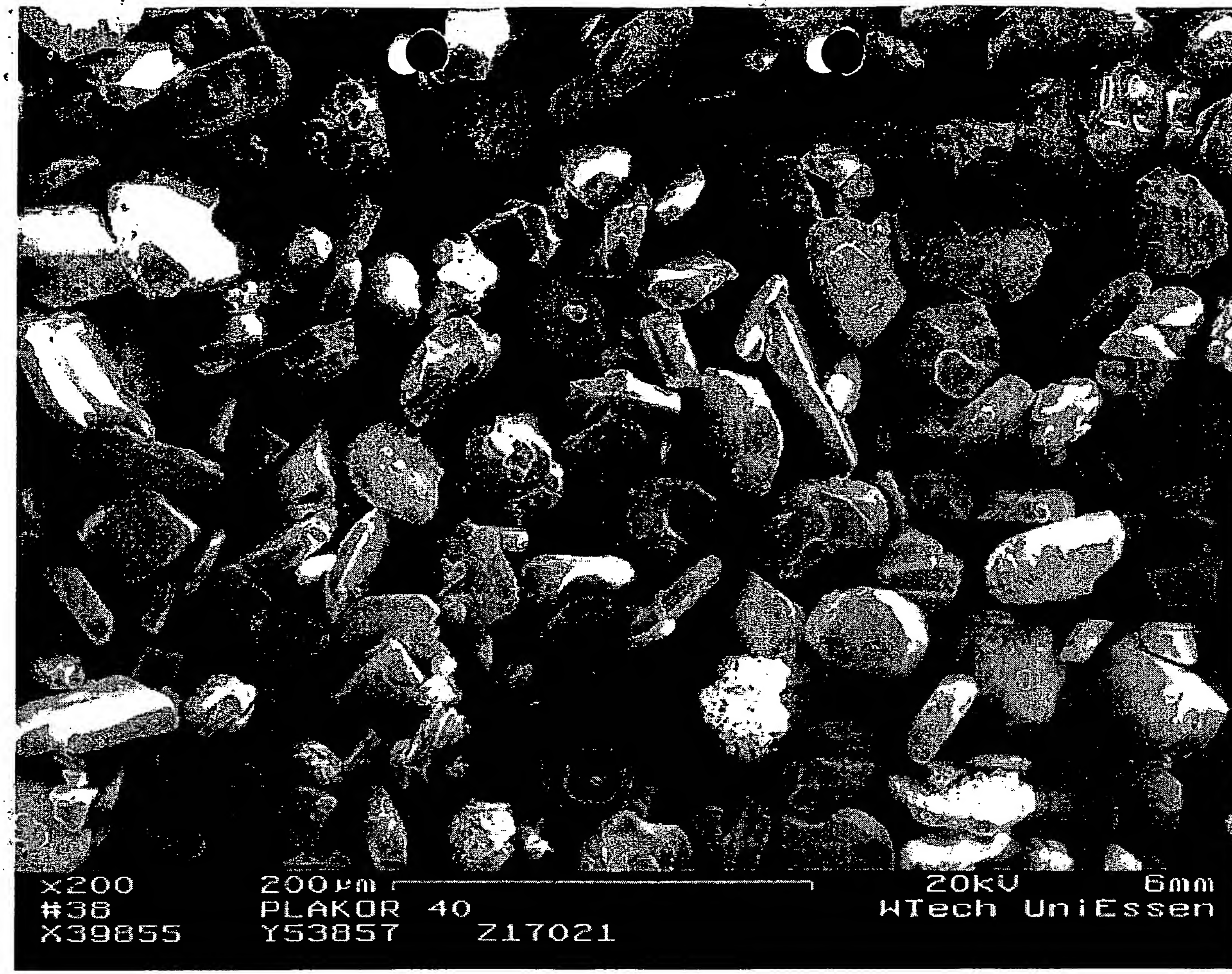
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WTech UniEssen
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1998 TAPPI PROCEEDINGS

PLASTIC LAMINATES SYMPOSIUM

August 17-20, 1998
Atlanta, GA
Hyatt Regency Hotel

COMPOSITE PANEL ASSOCIATION

LAMINATING MATERIALS ASSOCIATION

PRODUCING HIGH QUALITY THERMOFUSED MELAMINE PANELS.

R n Ohnesorge
Production Manager
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Stevens Industries Inc.
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ABSTRACT

Producing high quality thermofused melamine panels requires more than simply fusing a melamine saturated decorative paper to an industrial grade substrate. This report will look at some of the quality characteristics that the raw materials must possess, discuss control of the laminating process itself and review a few quality control / assurance procedures. The combination of these issues is important in producing high quality thermofused melamine panels.

INTRODUCTION

I will cover this topic by first reviewing the materials that comprise a thermofused melamine panel, the saturated decorative paper and the substrates to which they are pressed. We will look at some of the necessary characteristics these materials must possess in order to produce a high quality laminated product. We will review the laminating process and finishing by looking at several quality control / assurance tests, both in-process and follow up procedures that can be performed to ensure product quality.

MATERIALS

Thermofused melamines are normally pressed to industrial grade particleboard or medium density fiberboard using melamine impregnated or urea filled / melamine coated decorative papers. However, some specialty substrates such as moisture retardant or fire retardant are occasionally specified. Surface characteristics of the substrate are very critical. Today's marketplace is moving toward more exotic colors and prints, some of which typically end up on what could be refered to as a critical color list. These colors are very demanding of the substrate and have little or no ability to cover up any defects present on the surface of the core.

SUBSTRATES

One of the most critical attributes of the core is a pit free surface. Pitting on the surface will almost always show through these papers as a salty appearance on the laminated panel. A pit in the surface that is in excess of $1mm^2$ in diameter and deeper than .003" will not be covered up, or filled in by these "critical papers." These pits ultimately end up as little white spots on the surface of the laminate.

Sanding quality is very important as well, all of the primary sanding marks must be cleaned off the surface of the substrate, as these papers will not cover them up either. They will, as well turn into little white streaks on the surface of the finished product.

Among the defects that are caused by the substrate, low spots in the surface of the substrate continue to be one of the largest contributors to down graded panels. In the past these were primarily caused by a large piece of cured resin that managed to get into the surface of the panel during the forming process. After sanding and cooling down, the resin spot will shrink and / or crack. Recently, with new forming technology within the particleboard manufacturing arena, this defect has been reduced and most low spots are due to dents and depressions left on the surface of the substrate after the manufacturing process. In either case, a low spot in the surface of the core will turn in to a white spot in the surface f the laminated panel.

V

Another issue pertaining to the substrate is warp. Alth ugh there are many causes for warped raw material, most of which bring on spirited discussi n between supplier and customer, the fact remains that it causes manufacturing problems in the laminating process. Processing warped raw material requires other than normal operating profiles, which may lead to improperly cured laminates. Panels that are not properly cured could end up causing manufacturing problems in the customers mill room. And even if the laminator is successful in flattening the warped raw material during the pressing process, the panels may not necessarily remain flat. When the customer begins to machine the material, it may return t its original form once the stress in the panel is relieved by cutting or routing.

One of the most common problems causing warped raw core is an unbalanced Verticle Density Profile (V.D.P.). This occurs when there is more density on one face of the core than the other. Another is unbalance due to differing laminates from top to bottom face. This will be discussed later.

TREATED PAPERS

The decorative papers used are either solid colors, printed woodgrains or printed abstracts of some type that are saturated with melamine resin. The base paper itself can be specifically made either for fast cycle, low pressure laminating or a paper that is used in the manufacture of high pressure laminates. These papers typically range from 80 to 115 grams per square meter, with an overall treated weight of 200 to 350 grams per square meter.

The impregnating of the paper is a critical aspect as well. There are several specific areas of note that affect the flow of the resin. Some of these are resin percentage, flow percentage and volatile content. Consistent and controllable flow of the resin is of utmost importance in producing a melamine panel that meets industry expectations from a performance standpoint.

Aside from the aesthetic problems that arise from improper flow of the resin, a phenomenon also known as pitting can occur in the surface of the laminate due to improper treating. This typically happens when the resin has been dried, or "B-staged" too far, not allowing the resin to seal up properly during the pressing process. This will leave very small, microscopic pits in the surface of the laminate, which pick up and hold dirt; therefore, this phenomenon will not allow the product to have a cleanable surface. This type of pitting, and pitting in the surface of the core, are not related defects, and do not look at all similar in the laminated panel.

Coordination between the specifications of the dry paper, the performance characteristics of the resins, and the capabilities of the press line requires a close relationship between the treated paper supplier and the laminator.

LAMINATING PROCESS

There are several different designs of fast cycle melamine press lines, side clamping carriages, front clamping carriages, and continuous double belt presses. The pressing operation which we will be discussing is the sid clamping type.

The pressing process will have an under pressure time that can range from as low as 17 seconds to as long as 40 seconds depending upon the type and base of the decorative paper being pressed. The pressing pressure ranges from 280 p.s.i. to 400 p.s.i with peak temperatures reaching, at times, 335°F. Following the actual pressing cycle, the panels are inspected on both faces for defects that would cause them to not be a 100% "A" grade panel. Some of the more typical types of defects would be the core related defects mentioned earlier as well as flaws in the paper or from the process such as laminate chips, dirt in the paper, cracks or holes in the paper and other foreign material that can enter in to the process. The panels are then stacked in "A" grade and "B" grade units prior to packaging.

WARP CONTROL DURING LAMINATING

7

[redacted]

Aside from the possibility of raw panels being warped and the need to flatten them ut during the pressing process, laminating panels that are unbalanced from face to back du to the paper, is the single largest factor contributing to warp. Unbalanced means having a face laminate and back laminate that are n t the same and do not have similar overall treated weights. This issue typically stems from the use of base papers that have been made for the manufacture of High Pressure Laminates. However, it can, at times be an issue with papers designed for Low Pressure laminating.

When calculating the treated weight of a paper, the basis weight of the paper prior to treating is divided by the inverse of the resin treatment percentage. This gives the overall weight of the treated paper.

Example:	80 gram dry paper
	resin treatment 60% (.60)
total wt =	80 ÷ .40 (40%) = 200gms/m^2

The above method will allow a laminator to calculate the overall weight of all treated papers being laminated; therefore, allowing for more consistent warp control when not making panels with the same laminate on both faces. The higher the weight of the paper the more it will shrink during post cure causing the warp to occur.

When setting the guidelines for the allowable difference from face to back in overall weight, a laminator must also consider the thickness of the substrate that is being laminated. The thicker the core, the greater the difference in weight from face to back that can be allowed while still using normal operating parameters. For example, if the core thickness is .745 inches, a difference in paper weight of 20 gms/m^2 could be allowed without adjusting press conditions. If the core thickness is 1.120 inches, a difference in weight of 35 gms/m^2 could be allowed without requiring condition adjustments. Laminates on thinner cores should be kept as close as possible to equal weights in order to keep from producing warped panels.

Also, when considering the treated papers for panels that are not two sided (same color on both faces), it is not wise to laminate 100% melamine papers on one face with urea / melamine papers on the other face. The melamine face will have a tendency to shrink more than the urea / melamine face.

QUALITY CONTROL / ASSURANCE TESTING

During the pressing operation, there are quality assurance tests that a laminator can perform to ensure that the proper level of cure is being put into the product. During the pressing operation, it is a good idea to verify the operating temperature at the board paper interface. The operating temperature is the peak temperature reached during the under pressure time of the pressing cycle. This is done by placing a thermocouple wire between the board and the paper and allowing it to go through the pressing cycle. This should not be done until the press has reached equilibrium. The following cure tests should be performed shortly after the operating temperature has been verified.

The first cure test is a 2 minute formic acid test. This evaluation of cure is to be performed on a sample from a panel that is selected once the press has reached its steady state of equilibrium. Once a panel has been targeted for testing, 3 solutions of formic acid in strengths of 17%, 25% and 33% are to be placed on the surface of the laminate within 2 minutes of it exiting the press. Three or four drops of each solution, covering approximately 1 or 2 cm^2 should be placed on the surface of the panel. The acid is to remain on the panel, undisturbed for a period of 2 minutes. After 2 minutes, the acid solutions should be wiped clean with a dry cloth, followed by cleaning the test area thoroughly with a damp cloth, and again drying the test area. Evaluation of the test area should be done 5 minutes after cleaning the solution off of the laminate surface. Following the evaluation, the press operator can adjust the pressing parameters, should the results indicate so, to enable the process to put the optimum level of cure on the panels.

The formic acid test should be followed by the 20 minute boiling water test. This test should not be performed on a laminate that is still hot. Allowing the sample to cool down for 30 minutes is quite sufficient. After cooling, bring a cup of water to a boil, splash a little f the water on the surface of the laminate. Place the hot cup on the laminate in

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